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Sonocatalytic degradation of Direct Blue 71 azo dye at the presence Zero-Valent Iron (ZVI)

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ABSTRACT

The sonocatalytic degradation of Direct Blue 71 (DB71) azo dye has been studied in the presence of low-frequency ultrasound (20 kHz). An initial concentration of 50 mg L^{-1} of dye, within the range of typical concentration in textile wastewaters, was used. Batch sorption studies were conducted to study the effects of various parameters such as Zero-Valent Iron (ZVI) dose, pH, and effect of different power levels (20, 55, and 95 W), on DB71 oxidation. Oxidation of DB71 with the assistance of ultrasound was enhanced with the increase in ZVI initial concentration and ultrasonic power, and with the decrease in pH. It was observed that the color removal efficiency was influenced by the solution H_2O_2 amount. Accordingly, removal of 91.2% color and 97.2% total organic carbon of dye was achieved by applying the optimal operational parameters with 0.3 g L^{-1} of catalyst, 2.5 pH and 25°C, during 20 min. In hybrid study, the color removal yield of 100% was obtained by combination of US/ZVI/H_2O_2 at dyestuff concentration of 100 mg L^{-1}. The amount of catalyst and power levels had an important effect on the color removal yields ($p < 0.05$).

Keywords: Azo dye; Direct Blue 71; ZVI; Oxidation; Ultrasound; TOC

1. Introduction

Decolorization of wastewaters is one of the significant problems as the dye will be visible even at low concentration. Azo dyes constitute the largest class of dyes and are widely used in a variety of industries from textile to cosmetics [1–3]. About a half of global production of synthetic textile dyes (7×10^5 t per year) are classified into azo compounds that have the chromophore of –N=N– unit in their molecular structure. These azo dyes are known to be largely nonbiodegradable in aerobic conditions and to be reduced to more hazardous intermediates in anaerobic conditions [4]. Thus, there is a need to remove dyes from wastewaters before discharging it to receiving waters. Serious environmental problems have led many researchers to treat textile wastewater using traditional treatment methods such as chemical coagulation followed by sedimentation, ozonation, adsorption, electrochemical oxidation, photo catalytic discoloration. Nevertheless, these methods were insufficient to treat azo dye wastewater with high concentration and chroma [5,6]. Therefore, it is urgent to seek for some novel technologies to decolorize or degrade these organic dyes pollutants. As an alternative method, ultrasonic irradiation aiming at degrading organic

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pollutants including dyes has received much attention [7]. Ultrasound is a viable advanced oxidation processes (AOPs) technique that generates $OH^{\bullet}$ radicals from water pyrolysis during the implosion of acoustic cavity bubbles [8].

Sonochemical reactions are induced upon high-intensity acoustic irradiation of liquids at frequencies that produce cavitation (typically in the range 20–1000 kHz). Cavitation is a phenomenon of micro-bubbles formation, and their growth and implosion in the irradiated liquid. The extreme temperature and pressure released during adiabatic bubble collapse causes the fragmentation of those gas molecules trapped in the microbubbles into radical species. These radical species can either recombine or react with other gaseous molecules within the cavity or in the surrounding liquid, after their migration [9]. As ultrasound alone is insufficient for total mineralization of organic compounds in water, the addition of various additives is of common interest for improving mineralization reactions [8].

The combination of ultrasound and Zero-Valent Iron (ZVI) is a good alternative to increase the efficiency of degradation process. Since ZVI is relatively inexpensive and nontoxic. Sonication in the presence of elemental iron has been explored for the reduction in hazardous compounds [10–13]. Application of ZVI together with ultrasonic irradiation leads to enhanced mass transport of reactants to the metal surface, where ultrasound increases the defects and the number of active sites while continuously cleaning it. A simplified reaction scheme occurring in water during sonolysis in the presence of ZVI (Fe^{o}) is the following [8]:))) = ultrasound.

$$H_2O+)))\rightarrow OH^{\cdot} + H^{\cdot} \quad (1)$$

$$OH^{\cdot} + H^{\cdot}\rightarrow H_2O \quad (2)$$

$$OH^{\cdot} + OH^{\cdot}\rightarrow H_2O_2 \quad (3)$$

$$Fe^{\circ}+)))\rightarrow Fe^{2+} + 2e^{-} \quad (4)$$

$$Fe^{2+} + H_2O_2\rightarrow Fe^{3+} + OH^{\cdot} + H^{-} \quad (5)$$

$$Fe^{2+} + OH^{\cdot}\rightarrow Fe^{3+} + OH^{-} \quad (6)$$

$$Fe^{3+} + H_2O_2\rightarrow Fe^{2+} + OOH^{\cdot} + H^{+} \quad (7)$$

$$Fe^{\circ} + Fe^{3+}\rightarrow 3Fe^{2+} \quad (8)$$

$$Fe^{\circ} + H_2O_2\rightarrow Fe^{2+} + OH^{\cdot} + H^{+} \quad (9)$$

To our knowledge, there was limited report on the degradation of Direct Blue 71 (DB71) azo dye by the oxidation via AFP in combination with ultrasound. Therefore, in the study, the influence of various additives such as ZVI and hydrogen peroxide (H_2O_2) on the sonolytic degradation of DB71 azo dye was investigated in company with ultrasound working at 20 kHz due to sonochemical work using the low-frequency ultrasound [14]. Additionally, the effects of some experimental parameters such as ultrasonic power density (20 kHz), ZVI addition levels, pH, and H_2O_2 on the sonochemical degradation of dye were studied. The mineralization of DB71 in terms of total organic carbon (TOC) removal was also explored.

2. Experimental

2.1. Materials

DB71, azo dye, was purchased from Aldrich and used as received. The molecule structure of DB71 is shown in Fig. 1 [15]. ZVI powder (analytical grade, 10 μm) was obtained from Merck. H_2O_2 (analytical grade, 30% w/w) was obtained from Riedel-de Haën.

2.2. Methods

The experimental setup for ultrasonic irradiation is shown in Fig. 2. It is consisted of a 250 mL glass cell surrounded by a water-cooling jacket ($T = 20 \pm 2$°C), a probe type transducer. A stock solution of DB71 was prepared fresh with de-ionized water before each run and the initial concentration (C_o) was kept at 50 mg L^{-1}. Sulfuric acid or sodium hydroxide was used to adjust the initial pH of the dye solution. After 250 mL stock solution was fed into a glass reactor, a given amount of H_2O_2 and ZVI was added into the reactor. The ultrasonic bath operated at 20 kHz and a

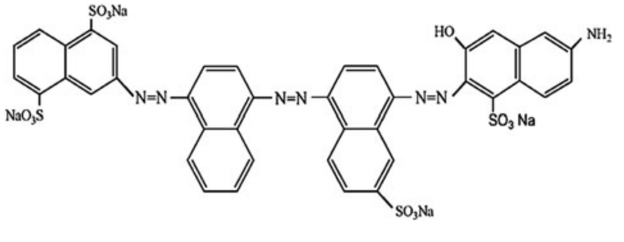

Fig. 1. Molecular structure of azo dye DB71.

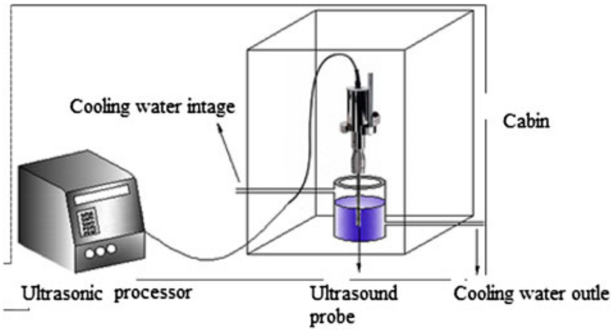

Fig. 2. Experimental setup.

US power of 750 W (Cole Parmer Ultrasonic Processor). The distance between the bottom of the reactor and the ultrasonic bath was maintained at 3 cm. The sonication was administered in a pulse mode of 20 min off. The reactor was immersed into a water bath to keep the temperature at 20°C. The experiment was carried out using different amplitude levels (30%, 60%, and 90%). The power level (Watts) was measured the by using Powmet-230 Power Meter. A total of 30%, 60% and 90% amplitude levels were measured as 20, 55, and 95 W, respectively.

The suspended ZVI particles were separated by filtering them through a 25-mm diameter, 0.45-μm filter. Color measurements were carried out in 587 nm wavelength which was determined as a result of spectrum analysis. The spectrum analysis, which was carried out in different concentrations, is given in Fig. 3. The DB71 concentration was measured using a spectrophotometer (UV-160A Shimadzu) at 587 nm. The mineralization of DB71 was identified by the reduction in TOC, as measured using an Apollo 9000 TOC-TN analyzer.

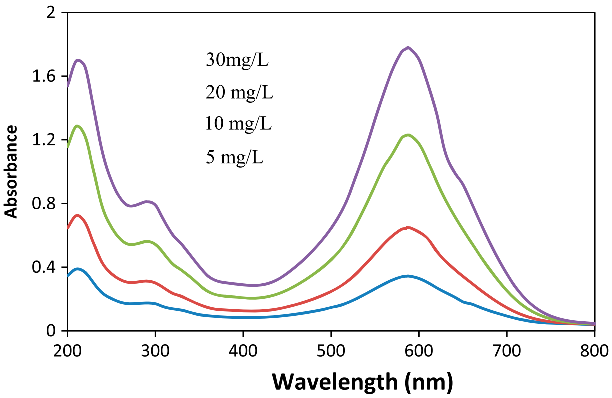

Fig. 3. UV–vis absorption spectra for DB71 dye.

2.3. Statistical analysis

Data obtained from analysis of the samples were evaluated statistically using analysis of variance (ANOVA) and the differences among means were compared with the Duncan's multiple range tests (using SPSS statistical software program version 17 (SPSS Inc., Chicago, IL, USA; SPSS 1999). Significance level of 95% ($p < 0.05$) was used for statistical differences, unless otherwise stated.

3. Results and discussion

3.1. UV–vis spectra

The maximum absorbance values of DB71 azo dyestuff were determined by scanning absorbance in 200–800 nm wavelengths. The maximum wavelength determined by absorbance measurements between 200 and 400 nm represents aromatic while the maximum wavelength determined by absorbance measurements between 400 and 800 nm represents visible region. Also, the wavelength, which color measurements were done was determined by using maximum peak obtained in visible region.

The obtained wavelength is similar to results in literature. The maximum peaks that were determined in UV region for the dyestuff have 210 and 290 nm.

DB71 is a three azo dye in which the chromophore part of molecular structure contains azo linkage and shows a strong absorbance in the visible region, while the absorbance peaks of the benzene and naphthalene rings are appeared in the UV region. The absorbance peaks at UV region (210 and 290 nm) and at 587 nm are, respectively, attributed to these aromatic rings and azo linkage [15,16].

3.2. Effect of ZVI on ultrasonic degradation of DB71 at different power levels

The substance additions which can increase degradation rate have been gradually common for mineralization reactions since the mineralization of organic compounds in water by ultrasound alone is not adequate. Such additions increase the formation of $^{\cdot}OH$ radical. In the study, color removal of DB71 dyestuff by ultrasound of 20 kHz accompanied ZVI was investigated in order to enhance the effect of ultrasound, and the effect of different ultrasonic power level was also determined.

The ultrasound assisted water splitting to produce H_2O_2 (reactions (1) and (3)) is considered to be the main advantage of combining sonolysis with sono-Fenton or sono-Fenton-like since the external addition of hydrogen peroxide is not required [17].

In order to determine the effect of ZVI on ultrasonic degradation of DB71, the aired $50\,\mathrm{mg\,L^{-1}}$ DB71 solution with and without ZVI was treated by ultrasound of 20 kHz at pH 2.5 and 20°C for 20 min. The effect of ZVI on ultrasonic degradation of DB71 by 20, 55, and 95 W power levels was indicated in Figs. 4–6, respectively, and Table 1.

As shown in Figs. 4–6, ultrasonic degradation of DB71 without ZVI is considerably slow in 20, 55, and 95 power levels while ZVI addition enhances removal yield. The catalyst significantly affected the degradation of DB71 ($p < 0.05$). In Fig. 4, removal yields of DB71 for 0.1, 0.2, and $0.3\,\mathrm{g\,L^{-1}}$ ZVI doses at the end of 20 min were found as 58.5%, 62.9% and 72%, respectively. This is account for the fact that the over dose iron would produce too many ferrous ions, which would scavenge $^{\cdot}$OH radicals via reaction (7). The addition of iron powder would improve color removal efficiently, and the decolorization rate increased with ZVI addition [7].

As shown in Fig. 5, the color removal of DB71 in power level of 55 W is more than 20 W. At the same time, the removal significantly increased as ZVI dose increased ($p < 0.05$). The color removal yields for 0.1, 0.2, and $0.3\,\mathrm{g\,L^{-1}}$ ZVI doses after reaction were found as 65.7%, 72.2%, and 82.9%, respectively. In other study, DB71 dyestuff was degraded by about 90% by laccase while ultrasound alone led to a complete decolorization after 23 h at 850 kHz [18].

The highest color removal yield was obtained by power level of 95 W. As shown in Fig. 6, the yield (48.7%) obtained by ultrasound alone rised to 64.7%, 73%, and 91.2% by addition ZVI of 0.1, 0.2, and $0.3\,\mathrm{g\,L^{-1}}$, respectively at the end of 20 min. The amount of ZVI had an important effect on the degradation of DB71 ($p < 0.05$). As can be seen from these results, additional ZVI and high power level increase degradability of the dye by accelerating the effect of ultrasonic reactions. The reason for this trend may be attributed to an increase in the number of active bubbles with increasing power delivered leading to an increase in the amount of $^{\cdot}$OH radicals produced [16]. The best color and TOC removal were obtained by ultrasonic frequency of 20 kHz, ZVI concentration of $0.3\,\mathrm{g\,L^{-1}}$ and 95 power level for 20 min. While azo dyes including DB71 have been decolorized and degraded by ultrasound working at 850 kHz within 3–15 h at 90 W and within 1–4 h at 120 W in other study, respectively [19]. Higher power level (95 W) increases effect of both homogenous and heterogeneous reactions due to enhancing the formation of cavitation. Consequently, it was proven that treatment of colored wastewater by US/ZVI system was an efficient method.

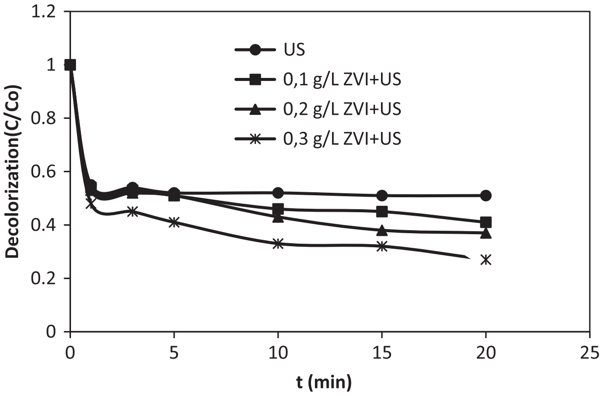

Fig. 4. The effect of ZVI on ultrasonic degradation of DB71 ($C_o = 50\,\mathrm{mg\,L^{-1}}$, pH 2.5, power = 20 W, $T = 20$°C, $t = 20$ min).

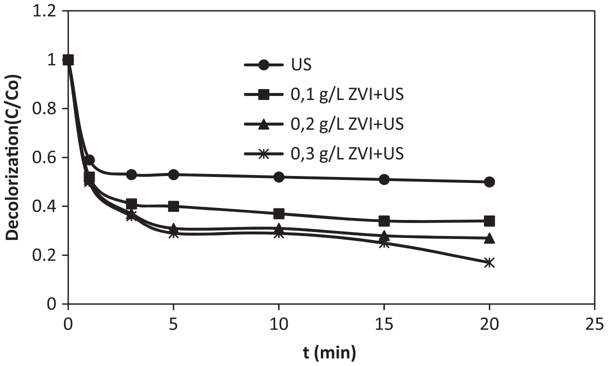

Fig. 5. The effect of ZVI on ultrasonic degradation of DB71 ($C_o = 50\,\mathrm{mg\,L^{-1}}$, pH 2.5, power = 55 W, $T = 20$°C, $t = 20$ min).

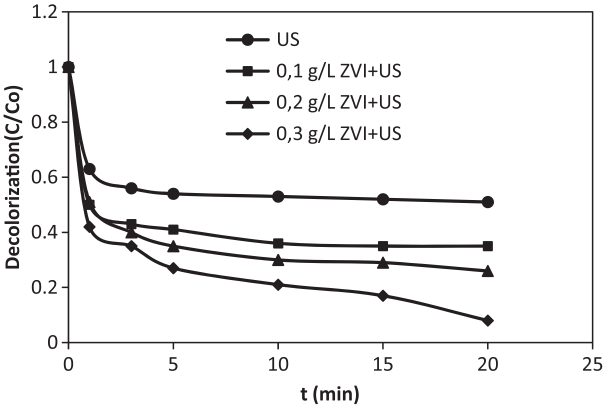

Fig. 6. The effect of ZVI on ultrasonic degradation of DB71 ($C_o = 50\,\mathrm{mg\,L^{-1}}$, pH 2.5, power = 95 W, $T = 20$°C, $t = 20$ min).

3.3. Effect of pH

Fig. 7 shows the effect of pH on sonochemical degradation of DB71. The results indicated that the

Table 1
Removal yields (%) by sonocatalytic degradation of DB71 at different power levels

	20 W	55 W	95 W
US	48.4^{a,A}	48.72^{a,A}	48.4^{a,A}
0.1 mg L^{-1} ZVI + US	58.5^{b,A}	65.7^{b,B}	64.7^{b,B}
0.2 mg L^{-1} ZVI + US	62.9^{c,A}	72.2^{c,B}	73^{c,B}
0.3 mg L^{-1} ZVI + US	72.0^{d,A}	82.9^{d,B}	91.2^{d,B}

$^{a\text{–}d}$Means in the same column followed by different lowercase letters represent significant differences ($p < 0.05$).

$^{A\text{–}C}$Means in the same line followed by different uppercase letters represent significant differences ($p < 0.05$).

ultrasonic degradation rates in strong acidic water (pH 2.5–3.0) are higher than those obtained in higher pH aqueous solutions (pH 4.0–5.5). The color removal yield was found as 91.2% at pH 2.5 while it was 19.5% at natural pH of the dye. There was no significant difference between pH 4 and 5 for the removal yields while the highest removal yield was statistically obtained at pH 2.5 ($p < 0.01$). This is due to the fact that iron powder is easily dissolved in acid solution producing ferrous ions, which has been proved by Liang et al. [20]. The produced ferrous ions react with H_2O_2 consisting as a result of ultrasound to generate $^{\cdot}$OH radicals. The resulting $^{\cdot}$OH radicals causes to degradation of more dye. In addition, $^{\cdot}$OH radicals could be produced at metal surface by the decomposition of H_2O_2, and a low pH favors the generation of OH$^{\bullet}$ radicals based on reaction (9) [1].

3.4. The change of TOC with ZVI

Degradation of the dye should be evaluated as an overall degradation process, involving the ultimate mineralization of both the parent dye and its intermediates. The most common way of estimating this overall process is to monitor the reduction in TOC [21]. Thus, in the ultrasound study in which optimum conditions were obtained, the effect of different ZVI doses on TOC was given in Fig. 8. The highest yield was observed as 97.2% for ZVI dose of 0.3 g L^{-1} in which color removal was also the highest while the TOC removal was 91.2% and 92.3% for ZVI doses of 0.1 and 0.2 g L^{-1}, respectively. Rapid mineralization of the simulated wastewater was achieved with high level removed at the end of reaction [22].

3.5. Effect of H_2O_2

H_2O_2 concentration is an important parameter for the degradation of the dye as a source of OH$^{\bullet}$ radical generation in the heterogeneous sonolytic reactor. In order to investigate the effect induced from the addition of H_2O_2, the sonolytic degradation of DB71 at 0–100 mg L^{-1} H_2O_2 was performed. These results are illustrated in Fig. 9. From the figure, it can be observed that increasing the dosage of H_2O_2 from 0 to 75 mg L^{-1} could enhance the decolorization of DB71 from 48.7% to 64.1% within 20 min of reaction. There was no significant difference between ultrasound alone and 50 mg L^{-1} H_2O_2/US treatment for the degradation of DB71 ($p < 0.05$). The highest color removal yield was obtained by 100 mg L^{-1} H_2O_2/US treatment while the lowest coor removal yield was obtained by 75 mg L^{-1} H_2O_2/US. The enhancement of decolorization of DB71 in this course is due to an increase in the $^{\cdot}$OH radical concentration. However, when further increase in the dosage above 75 mg L^{-1}, the decolorization of DB71 was not improved but dropped down (33.8%). Indeed, the decrease in the decolorization efficiency of DB71 at high dosage of H_2O_2 was mainly

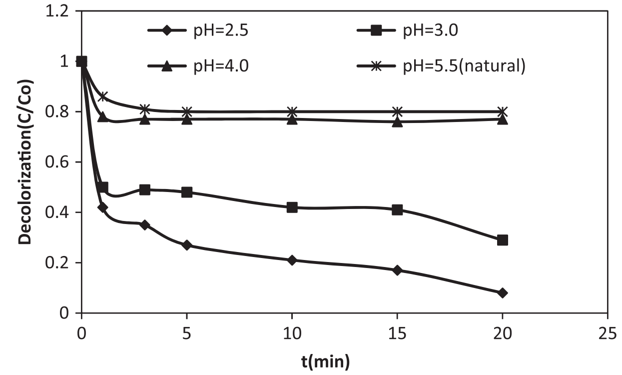

Fig. 7. The effect of pH on ultrasonic degradation of DB71 ($C_o = 50$ mg L^{-1}, ZVI = 0.3 g L^{-1}, power = 95 W, $T = 20$°C, $t = 20$ min).

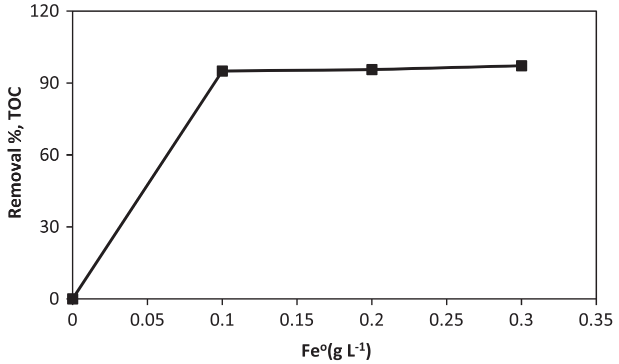

Fig. 8. Effect of ultrasonic degradation on TOC in different ZVI values of DB71 ($C_o = 50$ mg L^{-1}, pH 2.5, power = 95 W, TOC$_o$ = 135.6 **mg L^{-1}**, $T = 20$°C, $t = 20$ min).

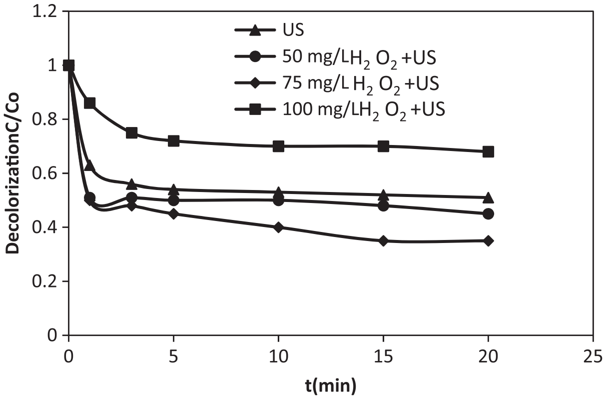

Fig. 9. The effect of H_2O_2 on ultrasonic degradation of DB71 ($C_o = 50\,mg\,L^{-1}$, pH 2.5, power = 95 W, $T = 20$°C, $t = 20$ min).

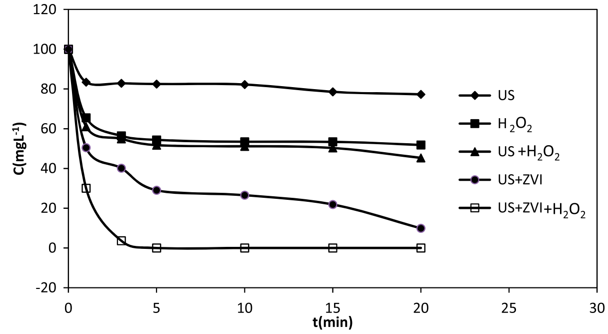

Fig. 10. The remaining dyestuff concentration by ultrasound oxidation of DB71 ($C_o = 100\,mg\,L^{-1}$, ZVI = $0.2\,g\,L^{-1}$, $H_2O_2 = 75\,mg\,L^{-1}$, power = 95 W, pH 2.5, $T = 20$°C, $t = 20$ min).

caused by the scavenging effect of excessive H_2O_2 to $^{\cdot}OH$ (Eq. (10)). In addition, the recombination of $^{\cdot}OH$ (Eq. (11)) also contributed for the declining of the decolorization efficiency of DB71 [23–26]. Therefore, an important step in the optimization of the method is the determination of the adequate amount of H_2O_2, to avoid an excess amount of reagent that can postpone degradation [27].

$$H_2O_2 + OH^{\cdot} \rightarrow HO_2^{\cdot} + H_2O \tag{10}$$

$$HO_2^{\cdot} + OH^{\cdot} \rightarrow H_2O + O_2 \tag{11}$$

As shown in Fig. 10, the color removal yield for dyestuff concentration of $100\,mg\,L^{-1}$ was 22.7%, 48.1%, 54.6%, 90.1%, and 100% by US, H_2O_2, US/H_2O_2, US/ZVI, and US/ZVI/H_2O_2, respectively. Generally, insufficient radicals are generated by ultrasonic irradiation, especially at low ultrasound frequencies. Therefore, the sonochemical process is often supplemented with other oxidants such as H_2O_2. Although H_2O_2 may act as a source of free radicals, the low volatility and the high solubility of H_2O_2 lead to a low H_2O_2 concentration in the cavitation bubbles. In addition, H_2O_2 also acts as the scavenger of the generated free radicals as $HO_2^{\bullet}$. Due to the low ultrasonic degradation rate of H_2O_2, it was tried to accelerate the decomposition of H_2O_2 via a Fenton-like process by heterogeneous catalysis including ZVI. Therefore, the presence of ZVI could increase decolorization rate significantly. However, the decolorization rate reached the maximum at ZVI dosage of $0.2\,g\,L^{-1}$. ZVI was corroded and thus Fe^{+2} (Eq. (4)) was obtained in US/ZVI system. The sonolysis degradation of DB71 increased significantly by the addition of Fe^{+2} (Fig. 10). It was showed that Fe^{+2} produced in US/ZVI system also can enhance the degradation of DB71. The highest color removal yield was statistically obtained by US/ZVI/H_2O_2 treatment ($p < 0.05$). Fe^{+2} is an effective oxidation catalyst for producing the organic radicals with reaction of free radicals and the organic substrate. Furthermore, Fe^{+2} can catalyze the production $^{\cdot}OH$ radicals with a sonocatalytic. Therefore, the presence of ZVI significantly increased the rate of decolorization [10,27].

4. Conclusions

The results obtained from this work showed that the combination of ultrasound treatment and catalyst could degrade DB71 effectively. The color removal yield significantly increased with the increase in H_2O_2 concentration, ZVI, and power levels, but decreased with the increase in initial pH value ($p < 0.05$). More ZVI dosages are required to achieve higher TOC removal. It was found that the highest color removal yield was obtained by US/ZVI/H_2O_2 combination. The effect of ultrasound increased with increasing ultrasonic power and additional oxidants. It can be concluded that ultrasound can be particularly useful for the treatment of solutions carrying dyes.

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